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(54) Title: LATEX COAGULATION IN A THICK FABRIC

(57) Abstract

The invention provides a fabric composite comprising a base fabric impregnated with a water based suspension binder such as latex, said fabric further comprising superabsorbent fibre distributed there within to expedite water emission from said binder to promote coagulation and film forming of the binder. The superabsorbent fibre is included between a lower limit at which the level of migration of the binder to the surface of the fabric is at an acceptable level for the percentage solid levels in the binder and an upper limit at which the fabric composite performance with respect to abrasion and strength is not reduced below acceptable levels and at which the superabsorbent fibre does not cause excessively rapid coagulation and absorption of the binder.

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Technical Field

- 5 The present invention relates to latex coagulation in fabric composites, in particular, but not exclusively, in so-called synthetic leather type materials. An example of such synthetic leather type material is the lining used within footwear e.g. Aquiline supplied by Texon UK Limited.

10 Background Art

- Typically, these types of fabric composite may be produced by impregnating a fabric base layer with a curable binder. In a broad sense the actual approach to manufacture is relatively simple in that the fabric is
15 presented to a bath of impregnant usually with some form of pressure to ensure adequate through penetration of the impregnant into the fabric. Typically, the fabric will be a non-woven needle felt.

- One method of manufacturing such fabric composites involves the
20 production of a thick needle felt, i.e. between 4-6 mm in gauge. This felt is impregnated with a soft rubber latex binder to increase its strength and abrasion resistance. The impregnated material is then split into several thicknesses i.e. 4 or more, of which each split thickness is required to have the same or substantially the same properties as the other
25 thicknesses. To achieve this, it is important that the distribution of binder within the full width or thickness of the fabric is substantially uniform. However, the binder is usually an aqueous suspension and, as such, is a liquid which has to be dried. In such drying procedures there is migration to the fabric surfaces leading to a lower binder concentration in the centre

of the fabric as compared to the surface. Thus there is a variation in performance through the gauge of the fabric composite and, typically, central splits of the fabric generally have a lower performance.

- 5 Previously, broadly three techniques have been used to mitigate the effects of binder migration in an impregnated fabric. The first technique involves chemical coagulation of the binder in a salt bath. Secondly, a heat sensitising additive can be included in the binder to stimulate heat coagulation of the binder which removes the requirement for a rinsing
- 10 stage (a more speedy coagulation reduces the time available for migration). Finally, in accordance with the third technique, the binder can be made more viscous by incorporation of appropriate additives and again such increased viscosity reduces the potential for migration. Each of these techniques involves additional problems in the fabric composite
- 15 manufacturing process. As will be understood by those skilled in the art, the first technique involves the necessity for extensive rinsing of the fabric composite to remove the salt used for coagulation. The second technique is not as effective as the first and relies on existing instability of the latex itself which it will be understood by those skilled in the art is not
- 20 consistent between batches of latex, may vary with age and certainly is dependent upon transient ambient conditions and more importantly does not work for many latex types. Finally, the third approach to reducing binder migration in composite fabrics is limited in that if the viscosity is too high the binder will not reach the centre of the fabric in an
- 25 appropriately short period of time without over pressurisation. Thus, the centre of the fabric may be less impregnated in any event without the effects of migration. In view of the above it is known to combine several of the above mitigation techniques during manufacture of the composite fabric. However, it will be appreciated that these migration mitigation

techniques significantly increase both the cost and complexity of the manufacturing process for these composites, because of the limitations on the type of latex which can be used, which will generally not be the cheapest, and the additional processing costs of salt coagulation. In addition, the expertise and time required to achieve the correct balance of chemicals for heat sensitising are significant. Furthermore, a compromise has to be made in achieving manufacturing efficiency between an acceptable degree of additional cost and retardation in the speed of composite fabric manufacture against adequate performance results for the various splits of fabric.

Summary of the Invention

It is an objective of the present invention to provide a composite fabric more readily manufactured to a consistent degree of performance for a much wider range of latex types.

In accordance with the present invention there is provided a fabric composite comprising a base fabric impregnated with a water based suspension binder such as latex, said fabric further comprising superabsorbent fibre distributed there within to expedite water emission from said binder to promote coagulation and film forming of the binder.

The composite may be suitable for a footwear lining.

Preferably, the fabric is a non-woven felt. The fabric may principally comprise either synthetic or natural fibres. Advantageously the base fabric may comprise polyester and/or polypropylene fibres.

- Advantageously the superabsorbent fibre may comprise fibres of a cross-linked acrylate co-polymer, partially neutralised to the sodium salt in fibre form. An example of a suitable superabsorbent fibre is that sold by Technical Absorbents Limited, England, under the trade name Oasis.
- 5 Alternatively the superabsorbent fibre may be based on salts of olefin/alkyl carboxylate copolymers, such as that sold by Camelot Superabsorbents, Netherlands, under the trade name Fiberdri.

- Alternatively the superabsorbent fibre may comprise a superabsorbent
- 10 fibre having a double layer structure, with the outer layer of superabsorbent material processed to an inner layer of acrylic fibre. An example of this type of superabsorbent fibre is that sold by Toyobo Company Limited, Japan, under the trade name Lanseal.

- 15 Further possible alternatives include superabsorbent fibres produced by covalent bonding of hydrophilic monomer chemicals onto the surface of fibres using free radical initiation or aqueous superabsorbent polymers based on alpha.beta-ethylenically unsaturated carboxylic acid monomer with softening monomers applied to fibre or fabric substrates as described
- 20 in US Patent No. 5 693 707.

- The superabsorbent fibre may be formed by pre-impregnating the base fabric with a superabsorbent solution in order to deposit a very thin layer on the fibres of the base fabric, thus forming a distribution of
- 25 superabsorbent fibres within the fabric.

Preferably the superabsorbent fibre is crimped as crimped fibres are less obvious on a split surface than fibres with no crimp.

Preferably the fabric composite comprises superabsorbent fibre between a lower limit at which the level of migration of the binder to the surface of the fabric is at an acceptable level for the percentage solid levels in the binder and an upper limit at which the fabric composite performance with respect to abrasion and strength is not reduced below acceptable levels and at which the superabsorbent fibre does not cause excessively rapid coagulation and absorption of the binder. It is also advantageous to use the minimum amount of superabsorbent fibre that produces acceptable performance in order to reduce cost as superabsorbent fibre is more expensive than traditional bulk fibres.

Preferably the composite fabric fibre content comprises at least 0.5% by weight superabsorbent fibre. Preferably the composite fabric fibre content comprises less than 10% by weight superabsorbent fibre. Advantageously the fibre content comprises between 1% and 6%, and preferably between 2% and 5%, by weight superabsorbent fibre.

In a preferred embodiment, the fibre content of a fabric composite according to the invention comprises, by weight, 65% polyester fibre, 30% polypropylene fibre and 5% Oasis fibre.

The binder is preferably a water suspension of polymer latex with pigment and extending filler.

The invention further provides a method of coagulation of aqueous latices in a fabric comprising the steps of distributing superabsorbent fibre within a base fabric, impregnating the base fabric with a water based suspension binder such as latex and allowing the wet fabric and binder combination to cure by coagulation of the binder within the fabric, the superabsorbent

fibre distributed within the fabric expediting water emission from said -
binder to promote coagulation and film forming of the binder.

The method may comprise the step of pre-impregnating a fabric with a
5 superabsorbent solution to produce a distribution of superabsorbent fibre
within the fabric by depositing a very thin layer on the fibres of the fabric.

Modes of Carrying out the Invention

10 An embodiment of the present invention will now be described by way of
example only.

In accordance with the present invention a fabric composite is provided
such that the latex binder coagulates soon after contact with the fibre and
15 before heat is applied to dry the composite. Such rapid coagulation or
film forming of the binder effectively prevents significant migration of the
binder at the surface with reciprocal diminution of the binder density in
central regions of the fabric.

20 Rapid coagulation of the binder is achieved by incorporating a low weight
per cent of a superabsorbent fibre. Examples of such superabsorbent
fibres include Oasis, produced by Technical Absorbents Limited of
England and Lanseal produced by Toyobo Company of Japan. Typically,
the superabsorbent fibre is incorporated up to a maximum weight
25 percentage of 10%, and preferably up to 5%, for several reasons including
such superabsorbent fibres being significantly more expensive than more
traditional bulk fibres, potential diminution in performance with regard to
abrasion and composite strength and problems with excessively rapid
coagulation and absorption of the binder. The superabsorbent fibres may

be added to a carded blend of 70% by weight polyester with 30% by -
weight polypropylene fibres.

The superabsorbent fibres enhance coagulation of the latex binder by
5 drawing the water from the binder into the superabsorbent fibre. Thus,
the liquid content of the latex binder solution is diminished and so the
latex is destabilised and coagulated. In the case of a soft rubber latex
binder, the effect of the superabsorbent fibre in the base fabric is to ensure
suitable coagulation of the binder for preparation of a so-called synthetic
10 leather material.

It will be understood that the inherent characteristic property of the
superabsorbent fibres are generally considered detrimental with regard to
impregnation due to the rapid absorption of liquid inhibiting penetration by
15 the coagulating binder. However, in accordance with the present
invention it has been noted that including superabsorbent fibres at a
reduced or low level in fact promotes coagulation of a latex binder
impregnant in a controlled way within the structure. In essence the
superabsorbent fibre sucks away water from the binder to cause it to
20 coagulate sooner after contact with a fabric and so removes or reduces the
necessity of incorporating chemical coagulants and thickening of binder
mixes.

It has been found that a weight percentage of 4% of Oasis superabsorbent
25 fibres evenly distributed throughout a non-woven fabric Oasis will
coagulate the following latices alone or along with a nitrile/PVC/calcium
carbonate binder solution:

Perbunan 2890 - Nitrile Rubber

Synthomer 7050 - Styrene Butadiene Rubber

Harco VV 530 - PVA

R & H Primal RA8 - Acrylic

Stahl RU3977 - Polyurethane

Wacker 424 - Ethylene Vinyl Acetate

5

Furthermore, 1% by weight Oasis superabsorbent fibre will coagulate Perbunan 2890, Harco VV 530 and Stahl RU3977, all without the need for any chemical coagulant, pH or viscosity modification. It has also been found that low solid content binders and low density fabrics i.e. lofty non-
10 woven fabrics have only limited variation on the effectiveness of the superabsorbent fibre effect in accordance with the present invention.

In order to illustrate the migration/coagulation of various latices with various levels of Oasis superabsorbent fibre, Table 1 below compiles for
15 each binder the degree of migration based upon a subjective quantised determination of the degree of migration. In the legend it will be seen that the degree of migration is given by zero for no migration to three for severe migration. It will be evident from Table 1 that inclusion of a relatively low weight percent of Oasis superabsorbent fibre radically
20 diminishes migration within the fabric composite.

Table 1

	<u>Latex Grade</u>	<u>Polymer</u>	<u>Oasis Level</u>			
			1 %	2 %	3 %	4 %
25	2890	NBR	0	0	0	0
	7050	SBR	1	0	0	0
	VV530	PVA	0	0	0	0
	HA8	ACRYLIC	2	1	0	0

RU3977	PU	0	0	0	0	-
EN424	EVA	1	1	0	0	.

0 = no migration

5 1 = patchy or slight migration

2 = more obvious migration

3 = severe migration

10 It will be understood by those skilled in the art that the proportion of solids within the binder is indicative of the volume of water within the binder and thus the effectiveness or ease of coagulation. Table 2 below illustrates migration/coagulation for Perbunan 2890 at various solid levels. Again it can be seen that it is at only very low i.e. 10% solids and very low Oasis level i.e. 1 % weight that there is severe migration and inclusion
15 of 2% Oasis superabsorbent fibre adequately reduces the level of migration to possibly acceptable levels.

Table 2

20	<u>% SOLIDS</u>	<u>Oasis Level</u>				
		1%	2%	3%	4%	5%
	10	3	1	1	0	0
	20	2	1	1	0	0
	40	0	0	0	0	0

25 In Table 3 two examples of a composite fabric in accordance with the present invention are disclosed. The base fabric is a non-woven felt comprising, by weight percentage, 65% polyester fibre, 30% polypropylene fibre and 5% Oasis superabsorbent fibre. This base fabric is impregnated with a binder without thickener, coagulant or ammonia and

in a dry binder to fibre ratio of 1 to 1, the materials are then dried and -
 cured as normal along with splitting and sueding to achieve a 0.7 mm
 gauge for the final fibre composite. In Example 1 the binder is a nitrile
 rubber/PVC/calcium carbonate dispersion including a colouring pigment
 5 whilst in Example 2 the binder is Perbunan 2890.

Table 3

		<u>BINDER</u>	
		<u>Example 1</u>	<u>Example 2</u>
		(NBR/PVC/whiting)	(100 % NBR)
<u>TEST</u>			
10	Weight (gsm)	284	405
	Gauge (mm)	0.66	0.70
	Tear (N) AL	18.7	38.1
15	AX	10.4	26.9
	300 Wet Abrasion	3/4mg SL.W	7/10mg SL/M.W

It will be understood by those skilled in the art that the fibre composite
 Examples 1 and 2 given in Table 3 are substantially suitable for use as a
 20 footwear lining material. However, it will also be understood that
 composite fabrics combining a base fabric layer whether woven or non-
 woven with an impregnated binder are used in a wide range of products
 and processes including linings for luggage, covering, packaging and
 possibly clothing. The present invention relates to a way of ensuring good
 25 coagulation or film forming of the binder whilst diminishing the possibility
 for binder solids migration to detrimental effect.

Fabric composites formed with a styrene butadiene rubber (SBR) latex are
 cheaper than traditional nitrile butadiene rubber (NBR) materials and it is

therefore advantageous to be able to use SBR based binders in making the fabric composites. In Table 4, two further examples using SBR binders are disclosed. The base fabric of Example 3 includes 8% by weight superabsorbent fibres and that of Example 4 includes 3% by weight superabsorbent fibres, with a binder to fibre ratio of 1:1.

TABLE 4

	BINDER COMPOSITION	WEIGHT %	
		EXAMPLE 3	EXAMPLE 4
10	Synthomer Bayer 76D40 Latex (74% butadiene SBR - self-crosslinking)	97.8	97.8
	Synthomer Bayer S30R Latex (30% butadiene self-crosslinking SBR)	32.6	32.6
15	70% Queensfil (calcium carbonate filler)	65.2	65.2
	BT338 (melamine formaldehyde crosslinking resin)	10.5	3.9
	Black PR (carbon black pigment)	6.5	6.5
20			

The performance results for Examples 3 and 4 are shown in Table 5.

TABLE 5

TEST	<u>BINDER</u>	
	<u>Example 3 (8% SAF)</u>	<u>Example 4 (3% SAF)</u>
Tear strength (N) - AL	10.9	18.5
- AX	12	18.1

Abrasion: weight loss (mg)	11.5	37.8
weight loss (%)	7.6	22.5
gauge loss (mm)	0.01	0.13

5 The composite fabric of Example 3 has good physical performance with good abrasion resistance but it also has an odour which will not be desirable for some uses. If the levels of superabsorbent fibre are reduced to 2%, all the odour is eliminated but the abrasion resistance is reduced. Fibre levels of about 3% with this SBR binder give acceptable abrasion
10 resistance with only a trace of odour. The odour has been found to be related to the crosslinking system of the SBR latex, particularly when based on formaldehyde, and the presence of superabsorbent fibres together. Some latices produce lower inherent odour, for example Bayer S44R.

15

The method of manufacturing the fibre composite is similar to that achieved previously. Thus, for example, the fabric is manufactured to an appropriate gauge and performance. As indicated above normally the base fabric will be a non-woven felt appropriately mechanically entangled to
20 achieve the necessary gauge and density. Once the base fabric is achieved it is impregnated in an impregnation bath with the selected aqueous binder dispersion. Typically, this impregnation process will be conducted under some form of forcing pressure such as nip rollers to stimulate and enhance impregnation and control the level of binder picked up by the fabric.

25 The now wet fabric and binder combination is immediately allowed to cure by coagulation of the binder within the fabric. Removal of the water will then be conducted typically by exposing the fabric to heat and/or blown air which may, additionally, be heated to stimulate drying. Once dry the fabric/binder fabric composite is normally split to form several

thinner gauge sheets of material. By the reduction in binder solid migration, the density of binder solids within each split sheet is more consistent and therefore performance of these sheets is also more consistent between splits of the base fabric/binder.

5

As indicated above, although the superabsorbent fibre presents significant advantages with regard to coagulation of the binder, care must be taken with regard to surface finish of the composite fabric. Superabsorbent fibres by their nature have different properties to general bulk fibres such as polyester or polypropylene and so when a significant percentage of superabsorbent fibre is added to the fabric it can create a blotchy surface appearance. Typically, this problem can be overcome by ensuring only the minimum proportion of superabsorbent fibre is incorporated within the base fabric. In addition or alternatively, the appearance can also be improved by the addition of sufficient dye or a soap such as Emulvin (TM) to colour the superabsorbent fibre and thus help colour distribution within the fabric and also by careful choice of pigment type. In particular some black pigments are of such a particle size that they will tend to accumulate within the superabsorbent fibre, producing a marbled effect.

20

Those skilled in the art will also understand that inclusion of superabsorbent fibres within a base fabric which is impregnated through an impregnation trough or bath can create an additional problem in that water uptake by the fabric as it passes through the impregnant trough or bath is significantly greater than with a simple bulk fibre i.e. polyester or polypropylene fabrics. Thus, the impregnant trough may become unexpectedly exhausted of water i.e. the binder solid to water ratio altered over a production run. Such variation in the impregnant proportions between binder solids and water obviously has significant effects upon the

impregnation capacity of the binder in that the more viscous binder - generally does not penetrate the fabric as quickly. In order to overcome this problem of water content diminution within the impregnant bath or trough when using a base fabric in accordance with the present invention it
5 may be necessary to regularly or consistently add water at a determinable rate to the impregnant trough or bath to ensure binder impregnant composition remains within desirable bounds. However, with very low levels of this superabsorbent fibre this will not usually be a serious problem.

10

The type of superabsorbent fibre used within the present invention will be determined by its effectiveness with regard to transportation of water and the end use of the composite fabric. It has been found that for shoe linings, where wear and durability are important, Oasis superabsorbent
15 fibre or Lanseal superabsorbent fibres have acceptable properties.

Typically, the fibres within the base fabric will be chosen in order to achieve the necessary feel for the fabric composite. Thus, for shoe lining material 1.7 decitex fibres may be used. Generally, the fibres have a
20 length in the order of 30 to 60 mm in order to ensure sufficient entanglement liaisons between fibres within a non-woven felt for good structural integrity. Additionally the level of crimping on the superabsorbent fibre affects the appearance of the finished product. The Oasis with no crimp can appear obvious on the split surface whereas the
25 Lanseal fibre with crimp is less obvious.

An advantage of the method of the invention is that it enables cheaper styrene butadiene rubber latices to be used instead of the nitrile butadiene rubber ones currently used for multiple split lining materials.

In an alternative arrangement, the core of the fibre composite may include superabsorbent fibres and the surface layers may be made of purely bulk fibre composition and in such a combination the binder solids may be
5 preferentially fixed in the core region of the fibre composite in comparison with the surface regions to achieve a more consistent binder solid concentration.

Alternatively the base fibre blend and fabric construction could be chosen
10 for some other application where subsequent impregnation with aqueous latex systems is required and where coagulation of the binder enhances the performance of the finished material, such as shoe insoles, filtration media, sound absorbing materials etc.

CLAIMS

1. A fabric composite comprising a base fabric impregnated with a water based suspension binder such as latex, said fabric further comprising superabsorbent fibre distributed there within to expedite water emission from said binder to promote coagulation and film forming of the binder.
2. A fabric composite according to claim 1 wherein the fabric is a non-woven needle felt.
3. A fabric composite according to claim 1 or claim 2 wherein the base fabric comprises polyester and/or polypropylene fibres.
4. A fabric composite according to any one of the preceding claims wherein the superabsorbent fibre comprises one or more of: superabsorbent fibres of a cross-linked acrylate co-polymer, partially neutralised to the sodium salt in fibre form; superabsorbent fibres based on salts of olefin/alkyl carboxylate copolymers; superabsorbent fibres having a double layer structure, with the outer layer of superabsorbent material processed to an inner layer of acrylic fibre; superabsorbent fibres produced by covalent bonding of hydrophilic monomer chemicals onto the surface of fibres using free radical initiation; or superabsorbent fibres produced by aqueous superabsorbent polymers based on alpha.beta-ethylenically unsaturated carboxylic acid monomer with softening monomers applied to fibre or fabric substrates.
5. A fabric composite according to any one of the preceding claims wherein the superabsorbent fibre is crimped.

6. A fabric composite according to any one of claims 1 to 3 wherein the superabsorbent fibre is formed by pre-impregnating the base fabric with a superabsorbent solution in order to deposit a very thin layer on the fibres of the base fabric, thus forming a distribution of superabsorbent fibres within the fabric.
7. A fabric composite according to any one of the preceding claims wherein the fabric composite comprises superabsorbent fibre between a lower limit at which the level of migration of the binder to the surface of the fabric is at an acceptable level for the percentage solid levels in the binder and an upper limit at which the fabric composite performance with respect to abrasion and strength is not reduced below acceptable levels and at which the superabsorbent fibre does not cause excessively rapid coagulation and absorption of the binder.
8. A fabric composite according claim 7 wherein the composite fabric fibre content comprises between 0.5% and 10% by weight superabsorbent fibre.
9. A fabric composite according claim 8 wherein the fibre content comprises between 1% and 6%.
10. A fabric composite according claim 9 wherein the fibre content comprises between 2% and 5%, by weight superabsorbent fibre.
11. A fabric composite according to claim 10 wherein the fibre content comprises, by weight, 65% polyester fibre, 30% polypropylene fibre and

5% superabsorbent fibres of a cross-linked acrylate co-polymer, partially neutralised to the sodium salt in fibre form.

12. A fabric composite according to any one of the preceding claims
5 wherein the binder is a water suspension of polymer latex with pigment and extending filler and possibly other additives.

13. A fabric composite according to claim 12 wherein the binder
10 comprises a latex of one or more of: nitrile butadiene rubber; styrene butadiene rubber; PVA; acrylic; polyurethane; or ethylene vinyl acetate, alone or along with a nitrile/PVC/calcium carbonate binder solution.

14. A fabric composite according to any one of the preceding claims
15 further comprising a dye or a soap to colour the superabsorbent fibre and help colour distribution within the fabric.

15. A method of coagulation of aqueous latices in a fabric comprising
the steps of distributing superabsorbent fibre within a base fabric,
impregnating the base fabric with a water based suspension binder such as
20 latex and allowing the wet fabric and binder combination to cure by coagulation of the binder within the fabric, the superabsorbent fibre distributed within the fabric expediting water emission from said binder to promote coagulation and film forming of the binder.

25 16. A method according to claim 15 wherein the method comprises the step of pre-impregnating a fabric with a superabsorbent solution to produce a distribution of superabsorbent fibre within the fabric by depositing a very thin layer on the fibres of the fabric.

17. A method according to claim 15 or claim 16 wherein the -
impregnation of the base fabric with the binder is conducted under a
forcing pressure to stimulate and enhance impregnation and control the
level of binder picked up by the fabric.
- 5
18. A method according to any one of claims 15 to 17 including the
further step of drying the fabric.
19. A method of manufacturing a composite fabric comprising the steps
10 of distributing superabsorbent fibre within a base fabric, impregnating the
base fabric with a water based suspension binder such as latex and
allowing the wet fabric and binder combination to cure by coagulation of
the binder within the fabric, the superabsorbent fibre distributed within the
fabric expediting water emission from said binder to promote coagulation
15 and film forming of the binder.
20. A method according to claim 19 wherein the method comprises the
step of pre-impregnating a fabric with a superabsorbent solution to
produce a distribution of superabsorbent fibre within the fabric by
20 depositing a very thin layer on the fibres of the fabric.
21. A method according to claim 19 or claim 20 wherein the
impregnation of the base fabric with the binder is conducted under a
forcing pressure to stimulate and enhance impregnation and control the
25 level of binder picked up by the fabric.
22. A method according to any one of claims 19 to 21 including the
further step of drying the fabric.

23. A method according to claim 22 including the further step of -
splitting the fabric/binder fabric composite to form several thinner gauge
sheets of material.
- 5 24. A method according to any one of claims 19 to 23 including the
step of adding a dye or soap to colour the superabsorbent fibre and thus
help colour distribution within the fabric.
- 10 25. A method according to any one of claims 19 to 24 wherein the
fabric is impregnated with binder through an impregnation trough or bath
and the water content within the impregnant bath or trough is monitored to
ensure the binder impregnant composition remains within desirable
bounds.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/02314

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 D06N3/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 D06N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
A	US 4 176 108 A (CAIMI RONALD J ET AL) 27 November 1979 (1979-11-27) page 1, line 8 -page 3, line 6; claims	1
A	US 5 763 524 A (ARKENS CHARLES THOMAS ET AL) 9 June 1998 (1998-06-09) abstract column 3, line 59 - line 67 column 8, line 31 - line 41	1

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex

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International Application No

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